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DEVELOPMENT OF PROPELLANTS  
CONTAINING AN ENERGETIC OXIDIZER**

**P. Rudy  
United Technology Center**

**TECHNICAL REPORT  
AFRPL-TR-66-186  
DECEMBER 1966**

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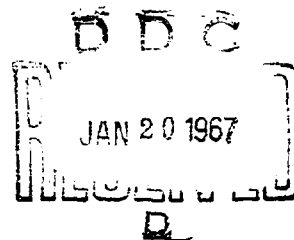
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EDWARDS, CALIFORNIA**



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## FOREWORD

(U) This second Quarterly Technical Progress Report under Contract No. AF 04(611)-10786 dated December 1966, covers work performed during the period 1 June 1966 through 31 August 1966, by United Technology Center, Sunnyvale, California. The contract was initiated under Air Force Rocket Propulsion Laboratory, Research and Technology Division, Project No. 3059. For internal purposes, this report is designated UTC 2139-QTR2.

(U) The Air Force program monitor is Karl W. Joffs, 1/Lt., USAF/RPMCP of the Research and Technology Division, Air Force Rocket Propulsion Laboratory, Air Force Systems Command, United States Air Force, Edwards, California 93523.

(U) The program manager for United Technology Center is E. J. Walden. Principal contributors to the work reported herein are:

J. W. Allan  
R. Alvarado  
L. S. Bain

T. W. Nakagawa  
J. T. Robinson  
M. E. Steinle

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## CONFIDENTIAL ABSTRACT

(C) United Technology Center (UTC) is conducting a 24-month program to produce the first practical propellants exploiting the high energy and favorable density of nitronium perchlorate (NP). During the present report period, it has been impossible to obtain Reta-coated NP, the form of the oxidizer which provides the most stable propellants. Emphasis, therefore, has been placed on definition and improvement of the storage stability of propellants containing uncoated, particulate NP. The effect of reduced storage temperatures on thermal stability of such a propellant has been determined. It has been found that a propellant which deteriorates to a useless and dangerous state in about 10 days at room temperature is stable for several months at 35° F. A still lower storage temperature (10° F) is less favorable. Optimization of the propellant formulation, particularly adjustment of crosslinker concentration, is expected to provide a further improvement in stability. Finely ground ammonium perchlorate may be used to increase solids loading and ballistic performance of the propellant without important effect on thermal stability. However, when deteriorated propellants containing high oxidizer loadings are ignited, they exhibit unusually violent explosive combustion.

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## CONTENTS

<u>Section</u>		<u>Page</u>
I	INTRODUCTION	1
II	THERMAL STABILITY	3
	1. Effect of Temperature on Propellant Stability	3
	2. Propellant A	5
	a. Effect of Storage at Constant Temperature	5
	b. Effect of Prior Storage at Low Temperatures	10
III	MECHANICAL PROPERTIES OF PROPELLANT A	13
IV	AMMONIUM PERCHLORATE AS A SUPPLEMENTARY OXIDIZER	15
V	CONCLUSIONS AND FUTURE ACTIVITY	21

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## ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	(U) Effect of Temperature on Compatibility and Cure	4
2	(U) Storage Stability of Propellant A (UTX-9118-1) at Constant Temperature	6
3	(U) Effect of Prior Storage at 35° F on Stability of Propellant A (UTX-9118-2) at 10° F	11
4	(U) Effect of Prior Storage at Low Temperatures on Stability of Propellant A (UTX-9118-3) at Elevated Temperatures	12
5	(U) Effect of Normal Burning of UTX-9118-4 63% Oxidizer, No AP	17
6	(U) Effect of Explosive Burning of UTX-9119-1 68% Total Oxidizer, 20% AP	18
7	(U) Effect of Explosive Burning of UTX-9120-1 73% Total Oxidizer, 22% AP	19
8	(U) Effect of Violently Explosive Burning of UTX-9121-1 78% Total Oxidizer, 23% AP	20

## TABLES

<u>Table</u>		<u>Page</u>
I	(U) Propellant Formulations	5
II	(U) Storage Stability of Propellants	7
III	(U) Mechanical Properties of Propellant A (UTX-9118-5)	13
IV	(U) Stability of NP/AP Formulations at 60° F	16



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## SYMBOLS

AP	ammonium perchlorate
DTA	differential thermal analysis
MRPX	a light, isoparaffinic oil obtained by special arrangement from Shell Oil Company
NP	nitronium perchlorate (C)
NTEB	nitrilotriethyl- $\beta$ -ethyleniminobutyrate, available from American Cyanamid Company
Reta	a poly(chlorinated xylene) coating developed by Union Carbide Corporation
UTREZ	$\alpha, \omega$ -dicarboxylated polyisobutylene

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## SECTION I

### INTRODUCTION

(C) Under Contract No. AF 04(611)-9894, UTC conducted a 12-month program to develop a curable binder containing a saturated hydrocarbon, difunctional polymer and to establish the compatibility of this binder with NP. A further objective of the program was ballistic characterization of the binder/NP formulation in small motors.

(C) These objectives were achieved by the development of a system based on UTREZ prepolymer, MRPX plasticizer, and NTEB crosslinker, (a tri-functional aziridinyl compound). Propellants containing both coated and uncoated NP of 30-40 mesh particle size were mixed, cast, and cured at room temperature. Those containing uncoated oxidizer were stable for only a few days at room temperature in a dry environment. Propellants containing Reta-coated oxidizer were found to be stable for periods up to 6 months under the same conditions. General characteristics of the propellants were: (1) insensitivity to impact and friction, (2) sensitivity to moisture, (3) limited thermal stability, and (4) reasonable mechanical properties.

(C) All of the propellants were ballistically characterized in micromotor firings, and a formulation containing Reta-coated NP was tested in nominal 1-lb motor firings. Efficient oxidizer utilization, high burning rates, and moderately high pressure exponents were indicated by these tests. The most significant relationships were believed to be those derived from the 1-lb motors:

$$r_b = 0.68 (P_c / 1,000)^{0.49}$$

$$K_n = 320 (P_c / 1,000)^{0.54}$$

(U) Under the present contract, experimental work is directed toward improvement of the basic propellant system with respect to stability, ballistic performance, and mechanical properties. Improved stability is by far the most important objective and is being emphasized accordingly.

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(C) Initial plans called for extensive work with Reta-coated NP. In February 1966, Union Carbide Corporation was in the process of preparing oxidizer for this program, when 6 lb of coated and partly coated material were destroyed in an explosion. This unprecedented event is still under investigation, and until safety of both the coating operation and the product is established, Reta-coated oxidizer will not be available for use under the present contract. Therefore, recent studies at UTC have been concerned with defining and improving the stability of propellants containing uncoated NP.

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## SECTION II

### THERMAL STABILITY

(C) Under the most ideal conditions, combinations of particulate NP with useful organic fuels ignite below 100° C, following extensive exothermic reaction which begins at materially lower temperatures. For example, the most stable propellant developed under the preceding contract contained Reta-coated oxidizer and exhibited thermal behavior comparable to that of the coated oxidizer itself. Under DTA, at a heating rate of 5° C/min, exotherm inception was typically 62 to 73° C and ignition occurred at 90 to 100° C.

(U) Since the propellant was mixed, cast, and cured at ambient temperature (i. e., only ca 35° C below the exotherm temperature), it is evident that the margin of safety was slim. This operation is akin to processing an AP-based propellant at temperatures above 400° F.

(U) By use of remotely operated equipment, it may be feasible to process at a relatively high temperature. However, it is unreasonable to expect the finished propellant to survive protracted storage under this condition. It is, therefore, appropriate to consider the increase in propellant life attainable by a rather modest reduction of storage temperature.

#### 1. EFFECT OF TEMPERATURE ON PROPELLANT STABILITY

(C) Figure 1 shows the effect of temperature on two processes: (1) gelation of UTREZ binder containing 25% MRPX plasticizer and 1.1 equivalents of NTEB crosslinker; and (2) the time required for visible gas formation when this binder formulation is in contact with uncoated, particulate NP. Considerable restraint must be exercised in the interpretation of these relationships. First, each curve is extrapolated on the basis of a simple process with invariant activation energy. Second, deterioration of the propellant is severe quite some time before gassing is observed. In short term studies, it has been found that the propellant may become ballistically useless before degradation is visible. Specifically, even when unconfined, the propellant may explode shortly after ignition, instead of burning in an orderly manner. Finally, gel time is plotted, rather than the time required for complete cure of the binder. Earlier it was speculated that the rate of degradation of the binder by interaction with NP might decrease following cure, since mobility of incompatible species would be reduced; however, this has not proved to be the case.

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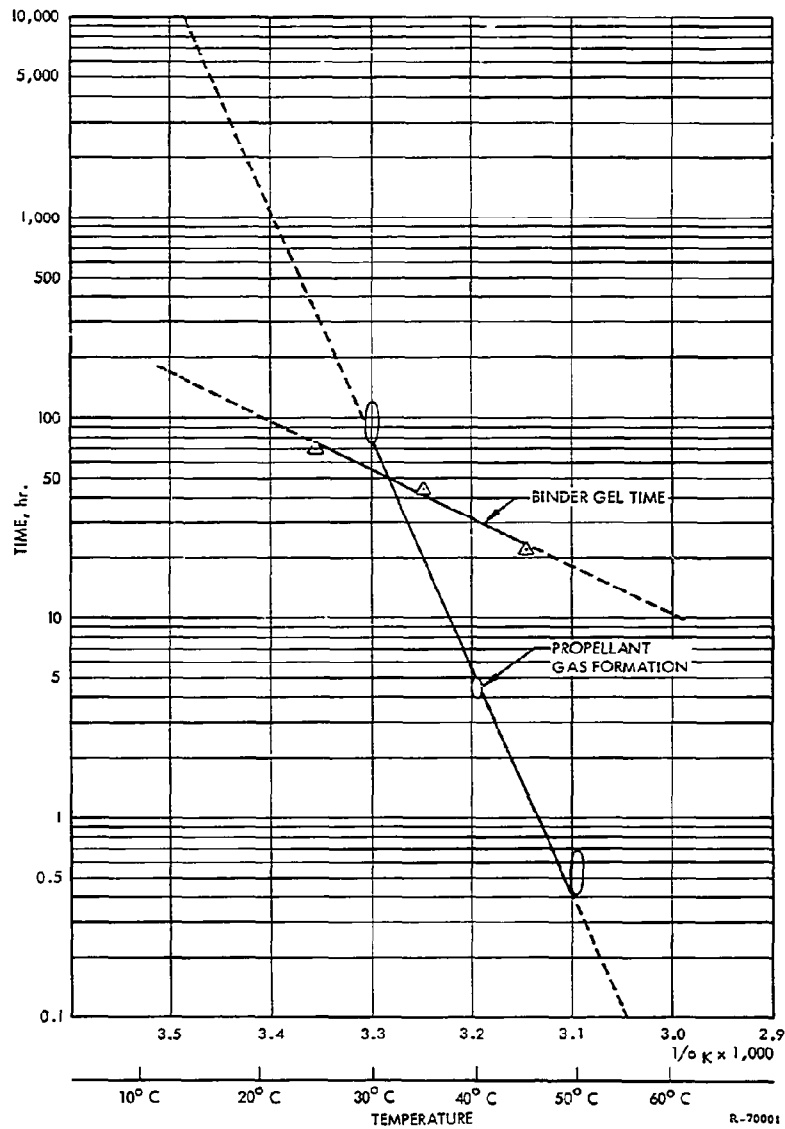


Figure 1. (U) Effect of Temperature on Compatibility and Cure

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(U) Even with the foregoing qualifications, there is every reason to expect a marked increase in propellant stability as storage temperature is decreased.

## 2. PROPELLANT A

(C) For initial studies, a well-characterized propellant formulation (Propellant A, UTX-9118) was selected (see table I). The binder formulation was optimized for use with Reta-coated NP, which interacts far less with the aziridinyl crosslinker than does the uncoated oxidizer.

(U) Samples of propellant were prepared in a dry box (moisture content less than 5 ppm) and stored at ambient or reduced temperatures within the same dry box. While samples in the individual refrigerated boxes remained at constant temperatures (60° F, 35° F, and 10° F), the samples at ambient were subject to unavoidable temperature variations. In general, ambient temperature averaged 70 to 75° F.

### a. Effect of Storage at Constant Temperature

(U) Propellant A was mixed and stored for 3 days at ambient temperature before samples were placed in storage at lower temperatures. Samples were removed periodically and subjected to DTA. Resulting data are given in table II and are plotted in figure 2. It is evident that reduction of storage temperature greatly improves stability of the propellant.

TABLE I  
(U) PROPELLANT FORMULATIONS

	Formulation UTX-			
	9118	9119	9120	9121
Oxidizer				
NP, 30-40 mesh, % w	63	47.6	51.1	54.6
AP, ground, % w	---	20.4	21.9	23.4
Aluminum, 40 $\mu$ , % w	2	2	2	2
Binder, % w	35	30	25	20
UTREZ, equivalent	1.0	1.0	1.0	1.0
NTEB, equivalent	1.2	1.2	1.2	1.2
MRPX, % w of binder	15	15	15	15

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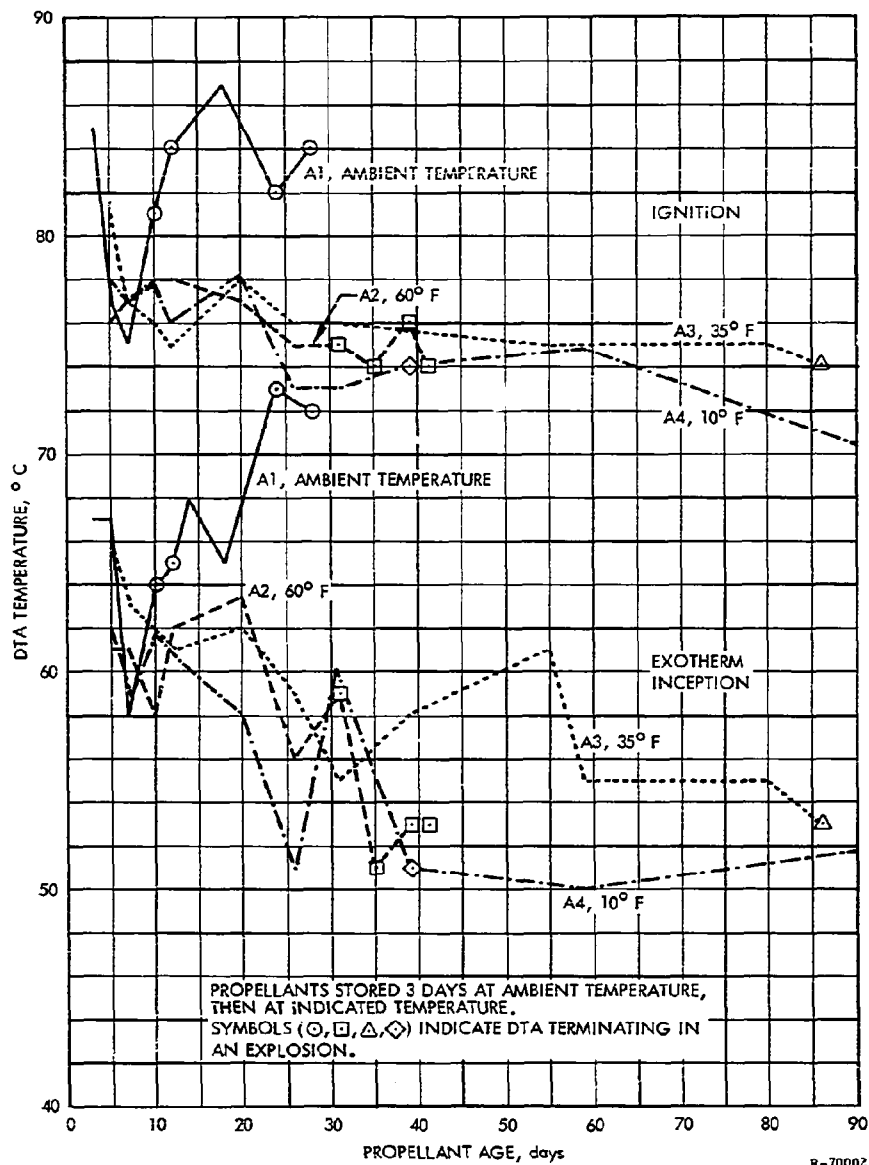


Figure 2. (U) Storage Stability of Propellant A (UTX-9118-1) at Constant Temperature

6  
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TABLE II  
(U) STORAGE STABILITY OF PROPELLANTS

Sample UTX-	A1 9118-1	ALT 9118-5	A2 9118-1	A2T 9118-5	A3 9118-1	A4 9118-1	B1 9118-2	B2 9118-2	C1 9118-3
Storage history, days/temperature	-/ambient	-/ambient*	3/ambient -60° F	7/ambient* -60° F	3/ambient -35° F	3/ambient -10° F	3/ambient -35° F	3/ambient 9/35° F -10° F	-/10° F
DTA, Exotherm/ignition ° C, Ago, days									
3	67/85	---	---	---	---	---	---	---	75/81
5	67/77	---	61/76	---	66/81	62/78	60/80	---	63/77
6	---	66/78	---	---	---	---	---	---	---
7	58/75	---	61/77	---	63/77	59/77	62/77	---	66/77
8	---	61/75	---	---	---	---	---	---	---
9	---	60/75	---	---	---	---	---	---	---
10	64/81X <sup>1</sup>	60/79	58/78	54/75	62/76	62/78	62/76	---	67/78
11	---	59/74	---	---	---	---	---	---	---
12	65/84X	---	62/78	---	61/75	61/76	---	---	63/78
13	---	56/75	---	---	---	---	---	---	---
14	68/85	57/75	---	---	---	---	---	---	---
15	---	---	---	52/76	---	---	---	---	---
16	---	52/75	---	---	---	---	---	---	---
17	---	---	---	---	---	---	---	---	---
18	65/87	---	---	51/77	---	---	---	---	---
20	---	---	64/77	---	62/78	58/78	60/79	61/80	62/80
21	---	---	---	---	---	---	---	---	---
24	73/82X	---	---	52/76X	---	---	---	---	---
26	---	---	56/75	---	59/76	51/73	52/77	51/75X	59/77
28	72/84X	---	---	---	---	---	---	---	---
30	---	---	---	53/75X	---	---	---	---	---
31	---	---	59/75X	---	54/76	60/73	63/76	49/75	59/79
34	---	---	---	---	---	---	---	---	---
35	---	---	51/74X	---	---	---	---	---	---
38	---	---	---	---	---	---	---	---	---
39	---	---	53/76X	---	58/76	51/74X	55/76	48/71	---
41	---	---	53/74X	---	---	---	---	51/74	60/77
46	---	---	---	---	---	---	---	---	---
49	---	---	---	---	---	---	---	---	---
52	---	---	---	---	---	---	---	---	---
53	---	---	---	---	---	---	---	---	---
55	---	---	---	---	61/75	---	---	---	---
59	---	---	---	---	55/75	50/75	54/77	56/76	---
69	---	---	---	---	---	---	61/77	---	46/75X
ca 70	---	---	---	---	---	---	---	---	47/75X
ca 80	---	---	---	---	55/75	---	56/73	---	55/74
ca 90	---	---	---	---	53/74X	52/70	54/74	---	59/74
ca 100	---	---	---	---	50/73	---	55/72	---	55/79
ca 110	---	---	---	---	60/74	---	57/71	---	64/74

1



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69  
ca 70  
ca 80  
ca 90  
ca 100  
ca 110

Sample  
UTX-  
Storage history,  
days/temperature

DTA  
Exotherm/ignition  
°C, Age, days

	C2 9118-3	C3 9118-3	C4 9118-3	C5 9118-3	D 9118-4	E 9119-1	F 9120-1	G 9121-1
3	---	3/10° F	3/10° F	3/10° F	-/60° F	-/60° F	-/60° F	-/60° F
5	62/78	9/35° F	29/35° F	29/35° F	-/ambient	-/ambient	-/ambient	-/ambient
6	---	---	---	---	---	---	---	---
7	64/85	---	---	---	---	---	---	---
8	---	---	---	---	---	---	---	---
9	---	---	---	---	---	---	---	---
10	66/79	---	---	---	---	---	---	---
11	---	---	---	---	---	---	---	---
12	66/81	---	---	---	---	---	---	---
13	---	---	---	---	---	---	---	---
14	70/87	70/84	---	---	---	---	---	---
15	---	---	---	---	---	---	---	---
16	---	---	---	---	---	---	---	---
17	---	---	---	---	---	---	---	---
18	68/82	65/79	---	---	---	---	---	---
20	---	---	---	---	---	---	---	---
21	---	---	---	---	---	---	---	---
24	73/82	64/79	---	---	---	---	---	---
26	---	63/76	---	---	---	---	---	---
28	70/80	---	---	---	---	---	---	---
30	---	---	---	---	---	---	---	---
31	67/82	62/78	---	---	---	---	---	---
34	---	---	---	---	---	---	---	---
35	---	---	---	---	---	---	---	---
38	---	---	---	---	---	---	---	---
39	---	54/76	---	---	---	---	---	---
41	66/80	---	---	---	---	---	---	---
46	---	---	---	---	---	---	---	---
49	---	---	---	---	---	---	---	---
52	---	54/76X	---	---	---	---	---	---
53	---	---	---	---	---	---	---	---
55	---	---	---	---	---	---	---	---
59	---	---	---	---	---	---	---	---
69	66/81	---	---	---	---	---	---	---
ca 70	---	---	---	---	---	---	---	---
ca 80	60/78	---	---	---	---	---	---	---
ca 90	62/79	---	---	---	---	---	---	---
ca 100	60/74	---	---	---	---	---	---	---
ca 110	64/78	---	---	---	---	---	---	---

\* Ambient temperature was lower than the normal value of 70 to 75° F  
† X = explosion

2

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(C) The behavior of sample A1 (ambient temperature) is representative of earlier results with propellants containing uncoated NP. The temperature of exotherm onset and ignition drops precipitously and reaches a minimum in about 1 week, then rises to or above initial levels. Interestingly, impact sensitivity of the propellant remains essentially constant (at ca 25 kg.cm.) throughout this period. Explosive combustion first occurs at, or shortly after the occurrence of minimum exotherm temperature. Exotherm temperature usually provides a better indication of imminent explosive combustion than does the ignition temperature. In the case of sample A1, DTA was not performed between the 7th and 10th days. It is believed that still lower exotherms could have been observed during this period. As a rule, the exotherm temperature of this formulation decreases to at least 55° F before explosive combustion begins.

(U) Using age at the first occurrence of explosive combustion as a crude standard, the effect of storage temperature is: ambient, 10 days; 60° F, 31 days; 35° F, 86 days; and 10° F, 39 days. The effect of decreased stability at 10° F compared to 35° F is unexpected but real. The mechanisms responsible for this effect are unknown, but it appears that several competing processes are at work, and their temperature sensitivities differ.

(C) Before discussing the experiments designed to elucidate this irregularity, it is perhaps worthwhile to call attention to a phenomenon which is peculiar to samples stored at 10° F. Over a period of weeks, a white, cloudy material condenses on the internal surface of the glass beakers containing these samples. While the amount of condensate is too small to characterize, it resembles NP, in that it "smokes" when exposed to humid air, and it is a powerful oxidizing agent.

(C) Another unexpected effect of low temperature storage deserves comment. A sample (C3) of freshly prepared propellant stored for 3 days at 10° F and 9 days at 35° F was transferred to storage at 60° F. Within 1 week this sample cured well and showed good thermal stability without explosive combustion until the 52nd day after mixing. In contrast, when initial storage consisted of 3 days at 10° F and 29 days at 35° F and was followed by storage at ambient temperature, the sample (C4) failed to cure. Therefore, it appears that at 35° F a stabilizing process accompanied by crosslinker depletion occurs. This process may be NP-catalyzed homopolymerization of NTEB.

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## b. Effect of Prior Storage at Low Temperatures

(C) To explore the possibility of exploiting low temperature exposure to stabilize NP propellants, a number of storage tests were conducted without altering the propellant formulation.

(U) Figure 3 indicates that 9 days of storage at 35° F failed to improve stability during subsequent storage at 10° F. It is evident, however, that the propellant used in this series was inferior to the particular batch employed in the tests shown in figure 2.

(U) Figure 4 shows test results with the same formulation which was placed in storage at 10° F immediately after mixing. Therefore, these samples were spared the initial thermal exposure suffered by the propellants discussed above. After 3 days at 10° F, a portion of the propellant was placed in storage at 35° F; subsequently, samples were removed from 35° F storage and were placed in storage at 60° F and at ambient temperature.

(U) Comparing the results of this test series with those shown in figure 2, it can be seen that early storage at low temperature promotes stability. First, the sample (C1) stored continuously at 10° F remained quite stable until sometime between the 41st and 69th days. The sample (C2) at 35° F remained stable for the duration of the test (ca 110 days). The sample (C3) at 60° F did not exhibit explosive combustion until the 52nd day. Of course, these improvements in stability might be explained simply by the minimization of initial exposure to ambient temperature. However, the behavior of the sample at ambient temperature (C5) seems to show a beneficial effect of 32 days storage at reduced temperatures (3 days at 10° F and 29 days at 35° F). Following this storage, sample C5 survived at ambient temperature for 9 days with good thermal stability and did not explode until the 14th day. By comparison, sample A1, which was exposed to ambient temperature continuously after mixing, was stable for 7 days and exploded when tested on the 10th day. It is clear that the difference between A1 and C5 is not great, but it still must be recognized that the latter was 32 days old when ambient storage began.

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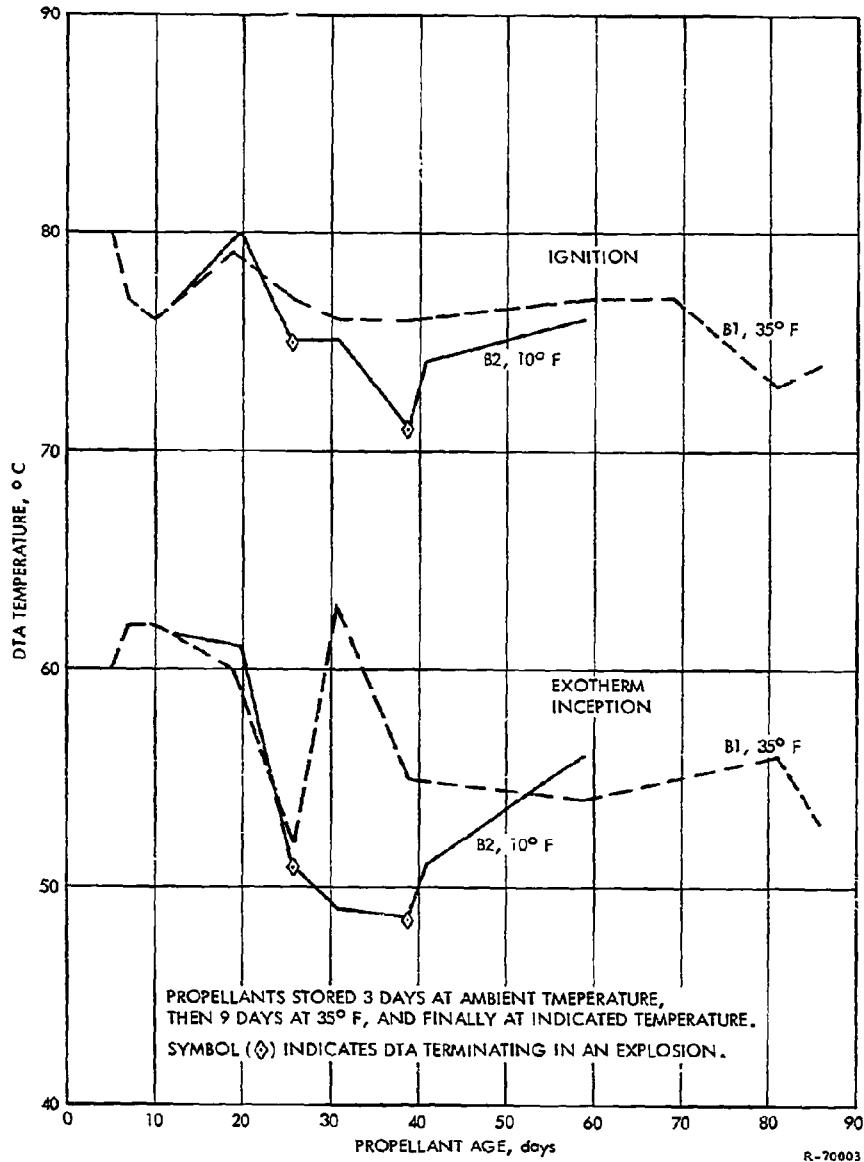


Figure 3. (U) Effect of Prior Storage at 35° F on Stability of Propellant A (UTX-9118-2) at 10° F

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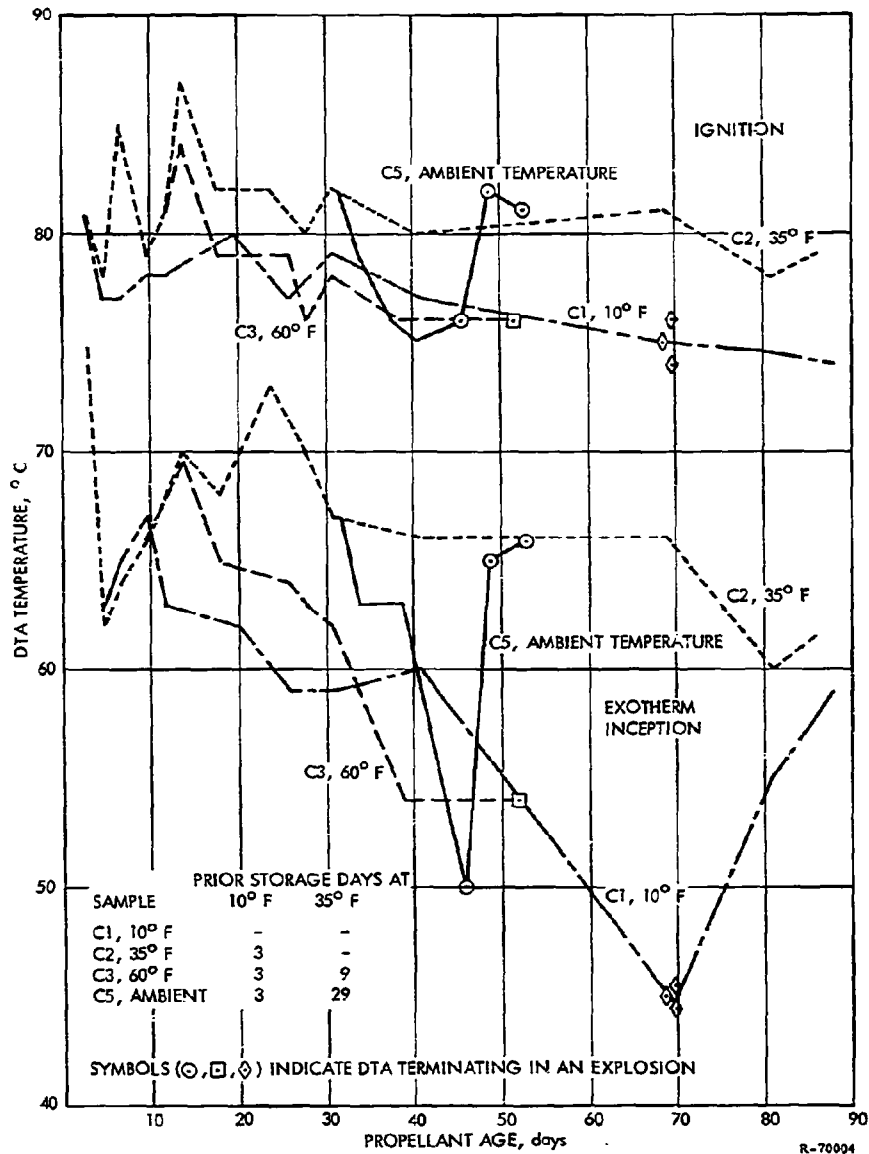


Figure 4. (U) Effect of Prior Storage at Low Temperatures on Stability of Propellant A (UTX-9118-3) at Elevated Temperatures

12  
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## SECTION III

### MECHANICAL PROPERTIES OF PROPELLANT A

(U) A limited study of the mechanical properties of propellant A was undertaken. Microtensile specimens were prepared, and tests were conducted as indicated in table III.

TABLE III

(U) MECHANICAL PROPERTIES OF PROPELLANT A (UTX-9118-5)

<u>Storage Condition</u>	<u><math>\sigma_m</math></u> <u>psi</u>	<u><math>\epsilon_m</math></u> <u>%</u>
8 days ambient temperature	7.4	25.9
8 days ambient temperature	9.3	29.0
10 days ambient temperature	5.1	18.0
10 days ambient temperature	9.1	28.7
13 days ambient temperature	Too weak to test	
7 days ambient, 8 days 60° F	7.4	20.4
7 days ambient, 8 days 60° F	9.3	26.6
7 days ambient, 10 days 60° F	9.8	17.8
7 days ambient, 10 days 60° F	11.0	30.5

Results of a parallel study of the thermal stability of this batch of propellant are shown in table II (A1T, A2T).

(C) Probably the only useful result of this exercise is the demonstration that the binder formulation employed does not cure properly in the presence of uncoated NP. With Reta-coated oxidizer, typical tensile properties are  $\sigma_m = 36$  psi,  $\epsilon_m = 43\%$ . It is expected that an increase in crosslinker concentration would provide materially better mechanical properties.

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## SECTION IV

### AMMONIUM PERCHLORATE AS A SUPPLEMENTARY OXIDIZER

(C) Limited studies have been conducted to determine whether finely divided AP can be employed to increase solids loading and specific impulse without impairing propellant stability. This approach has particular merit when Reta-coated NP is used, since particle sizes less than 30-40 mesh are not suitable for Reta coating. However, there may also be some value in systems containing uncoated NP. Since NP-binder interfaces are the site of the reactions leading to degradation, it may be desirable to limit this interfacial area by avoiding use of very finely divided NP.

(U) Samples containing various concentrations of AP and various solids loadings were prepared and tested, with the results shown in table IV. On the basis of this study, it appears that thermal stability is not importantly affected by the presence of AP. However, the tendency of the propellant to explode and the violence of explosion are increased at higher solids loadings.

(U) Some appreciation of the violence with which ca 2 g samples of the unconfined propellants explode can be gained by inspection of figures 5 through 8. These photographs show damage to the soft aluminum foil cups in which the test samples were ignited by use of a hot wire and a small piece of conventional AP propellant.

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TABLE IV

(U) STABILITY OF NP/AP FORMULATIONS AT 60° F

Sample UTX-	D 9118-4	E 9119-1	F 9120-1	G 9121-1
Binder, * % w	35	30	25	20
Al, % w	2	2	2	2
AP, % w	---	20.4	21.9	23.4
NP, % w	63	47.6	51.1	54.6

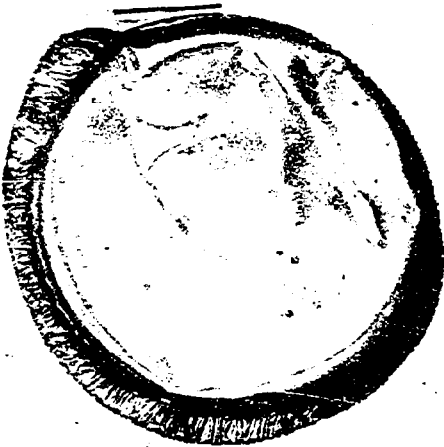
DTA  
Exotherm/Ignition, ° C  
Age, days

3	67/78	67/78	70/88	67/83
5	69/80	71/82	70/79	68/81
7	64/80	67/82	64/83	66/82
11	63/80	62/83	60/81	60/78
13	66/79	62/81	64/79	63/78
17	62/77	64/79	63/79	63/78
21	61/79	64/81	63/79X*	64/78
26	59/77	60/77X	63/77	57/76X
31	60/75	61/76	60/72	54/76X
34	59/75	61/77	58/75X	55/76X
41	50/74X	56/79X	59/75X	56/74X
Deflagration at 32 days	1 exploded 2 normal	2 exploded	2 exploded	2 exploded violently
Deflagration at 45 days	normal	exploded	violently exploded	violently exploded

\* X = explosion



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UTX 9118-4

DATE: 8-11-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 32 DAYS

R-70005

UTX 9118-4

DATE: 8-24-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 45 DAYS

5866-5

Figure 5. (U) Effect of Normal Burning of UTX-9118-4  
63% Oxidizer, No AP

17  
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UTX 9119-1

DATE: 8-24-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 45 DAYS  
5866-4

UTX 9119-1

DATE: 8-11-66

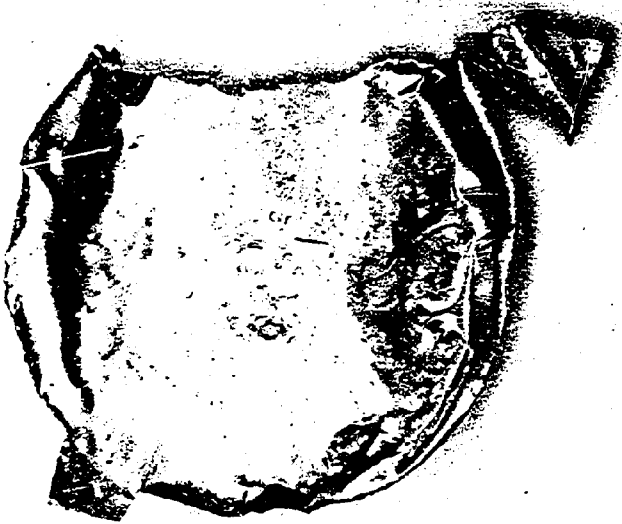
STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 32 DAYS  
R-70006

Figure 6. (U) Effect of Explosive Burning of UTX-9119-1  
68% Total Oxidizer, 20% AP

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UTX 9120-1

DATE: 8-11-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 32 DAYS

5866-3



UTX-9120-1

DATE: 8-24-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 45 DAYS

11-70007

Figure 7. (U) Effect of Explosive Burning of UTX-9120-1  
73% Total Oxidizer, 22% AP

19  
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UTX 9121-1

DATE: 8-11-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 32 DAYS

5866-2

UTX 9121-1

DATE: 8-24-66

STORAGE TEMPERATURE: 60° F

TOTAL DAYS CURE: 45 DAYS

R-70008

Figure 8. (U) Effect of Violently Explosive Burning of UTX-9121-1  
78% Total Oxidizer, 23% AP

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## SECTION V

### CONCLUSIONS AND FUTURE ACTIVITY

(C) Important increases in the life of propellants containing uncoated NP can be achieved by reduction of storage temperature. Of three reduced test temperatures, 60°, 35°, and 10° F, it appears that 35° F is the most favorable. Evidence obtained from storage tests suggests that a stabilization process accompanied by consumption of crosslinker occurs at 35° F. Increased concentrations of NTEB will be employed in future studies to enhance the stabilization process and still leave sufficient crosslinker for generation of acceptable mechanical properties during the cure.

(C) Finely ground ammonium perchlorate may be used to increase solids loading of propellant containing uncoated NP without important effect on thermal stability. However, when propellants containing high oxidizer loadings deteriorate, they exhibit unusually violent explosive combustion. Further work in this direction is not warranted until a more stable basic propellant system is developed.

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13. ABSTRACT (UNCLASSIFIED ABSTRACT)  A 24-month program is being conducted to produce practical, castable, composite solid propellants containing a high-energy oxidizer. Emphasis is being placed on improving the thermal stability and increasing the attainable solids loadings of propellants containing particulate oxidizer, a compatible binder (carboxy-terminated polyisobutylene), and polyfunctional aziridiny crosslinking agents.  During the present report period, emphasis has been placed on definition and improvement of the storage stability of propellants containing uncoated oxidizer. Reduction of storage temperature was found to extend propellant life. Maximum life was achieved at 35° F. Further reduction of the storage temperature to 10° F reduced propellant life. Increased concentration of crosslinker is expected to provide improved stability. Finely ground ammonium perchlorate has been employed to increase solids loading without materially affecting thermal stability. (U)		

## Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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HIGH-ENERGY SOLID OXIDIZER						
CARBOXY-TERMINATED						
POLYISOBUTYLENE						

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